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Mechanical properties, thermal degradation and natural weathering of high density polyethylene/rice hull composites compatibilized with maleic anhydride grafted polyethylene

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Abstract The influences of rice hull (RH) contents and particle sizes on the mechanical, physical, thermal and morphological properties of high density polyethylene (HDPE)/RH composites were investigated in the current research. Further study was conducted by incorporating maleic anhydride grafted polyethylene (MA-g-PE) to improve the interfacial adhesion between HDPE and RH. Experimental results indicated that the flexural and the tensile strengths were increased upon raising the silica-rich RH contents. With the addition of RH, the composites became brittle as was reflected by the lower impact strength. An improvement of all the flexural, tensile and impact strength was evident when the MA-g-PE was added in all levels. SEM micrographs revealed some RH pulled-out and numerous interfacial cavities occurred within the untreated HDPE/RH composites. With the presence of the compatibilizer, the interfacial adhesion was significantly improved. Lower water absorption was found when MA-g-PE was added in all compositions. Under UV exposure, the colors of the composites found to paler shade.

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Introduction

The global wood plastic composites (WPC) industry has experienced strong growth over the past few decades because of their several advantages over natural wood, such as low density, low cost, abundant availability, biodegradability and high specific strength and modulus [1–5]. The increasing demand for the use of natural wood has resulted in a serious problem of deforestation which consequently led to several natural disasters. This is why much of the current research concentrates on the development of materials that can substitute natural wood, such as WPC [1–9]. The WPC consists mainly of a polymer matrix, lignocellulosic filler such as wood flour and some additives [6]. The most commonly used matrices for WPC manufacture are thermoplastics such as polyethylene (PE) [3, 6, 7, 10], polypropylene (PP) [5, 9, 10] and polyvinyl-chloride (PVC) [1, 2, 4, 8, 10, 14]. The typical WPC produced in Asia mostly used poly(vinyl chloride) (PVC) as the matrix and wood flour as the filler [1, 15–17]. Western and European countries concentrated on utilizing PE and PP rather than PVC. In this research, high density polyethylene (HDPE) was selected as the matrix because of its wide applications and good processability. It is also inherently tough, flexible, nontoxic and odorless as well.

Recent developments of WPC using agricultural residues as reinforcing fillers is gaining extensive attention [2, 3, 5, 9–14]. Studies on particular agricultural residues such as straw [18–20], corncob [21, 22] and RH [2–5, 23] indicated their potential use as fillers; it has prompted some researchers to utilize RH in their research on the production of WPC. The fact that rice is cultivated massively in tropical countries means several million tons of RH is obtained every year. Yet,

only a minor quantity of this regularly renewable resource was utilized. A small portion is reserved for animal feed and the production of energy but a major portion is burnt in the field creating more CO_2 and additional environmental pollution. Conversion of these materials into useful industrial products would lead to alleviating waste disposal problems. So, RH was chosen as filler replacing the conventional wood flour to prepare WPC in the present study. Chemical analysis revealed that RH possesses chemical constituents closed to those of wood [2]. The RH is also biodegradable, inexpensive and has low bulk density, making it an attractive filler for WPC.

It is widely known that the mechanical and physical properties of WPC products are affected by a number of factors, such as the volume fraction and the aspect ratio of the fiber, fiber orientation, dispersion level, fiber–polymer adhesion, mixing time and processing temperature [1–23]. Another very important factor believed to also influence the mechanical properties of the composites is the compatibility between matrix and the filler phase. Hence, the incompatibility between the hydrophobic HDPE matrix and the hydrophilic RH filler is viewed problematic but it can be alleviated by incorporating an appropriate compatibilizer which is capable of enhancing the interfacial adhesion between the HDPE matrix and the RH phase [3, 23–25].

In the current research, the WPC prepared from HDPE and RH was investigated by studying the effects of RH contents and the particle sizes of the milled RH on the physical, thermal, mechanical and morphological properties of the HDPE/RH composites. Maleic anhydride grafted PE (MA-g-PE) compatibilizer was employed in order to promote the interfacial adhesion between the HDPE and the RH filler.

Experimental works

Materials

HDPE (HJ 1100, Bangkok Polyethylene) was selected as the polymer matrix. It has a melt flow index of 18 g/10 min and a density of 0.96 g/cm^3. MA-g-PE (Fusabond$^®$ WPC-576D, Dupont) was used as a compatibilizer. It has a melt flow index of 25 g/10 min and a density of 0.94 g/cm^3. RH was obtained mostly full-grained from Sookcharoen rice mill, it discarded from rice-milling process. It was grinded to fine particles by using a vibrational ball mill, and sieved to particles with average size of 75, 180, 250, 355 and 500 µm by using a vibrational sieve analyzer. RH particles of each size were dried in a hot air oven at 85 °C for 24 hours prior to produce WPC.

Preparation of the HDPE/RH composites

The experimental study was carried out over two parts. The first part involved a selection of the most appropriate content of

RH particles in the HDPE/RH composites for WPC applications. A series of HDPE/RH composites were prepared without any compatibilizer. The HDPE was firstly dry-mixed with various contents of oven-dried RH at 10, 20, 30 and 40 wt%. Then they were compounded and pelletized by using a twin screw extruder (Thermo HAAKE). The temperatures of the different zones during compounding were 140/160/160/160/150 °C, the die temperature was 150 °C which the screw speed was 50 rpm. The compound was obtained as filled HDPE pellets, they were then dried at 95 °C for 1 hr prior to injection molding (Manumold) at temperature of 180–190 °C to form specimens for subsequent evaluation of the properties of the composites. The second part concentrates on the influences of the MA-g-PE compatibilizer on the HDPE/RH composites. Another series of the HDPE/RH composites were prepared at the selected RH content but still using the RH of different particle sizes and different MA-g-PE contents. Aimed as an adhesion promoter, the MA-g-PE was employed at 1.0, 3.0, 5.0 and 7.0 wt% of RH. All the composites were prepared using identical processing conditions.

Testing and characterizations

The influences of the RH filler and the MA-g-PE compatibilizer contents and the RH particle sizes on the HDPE/RH composites were investigated by performing some mechanical tests namely impact, flexural and tensile tests at room temperature. The Izod impact test was conducted by using a pendulum type impact tester (Yasuda, 258PC) according to the conditions and the procedures described in ASTM D 256. Tensile test was conducted in accordance with ASTM D 638 with a cross head speed of 5 mm/min. Flexural test was performed using a 3-point bending mode following
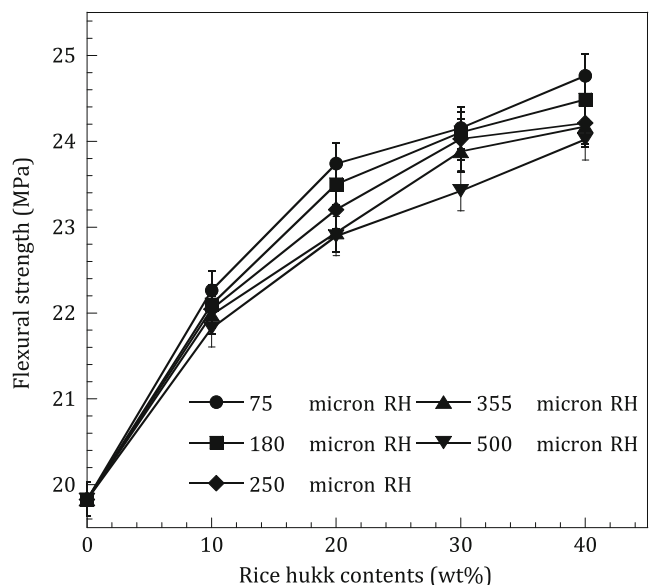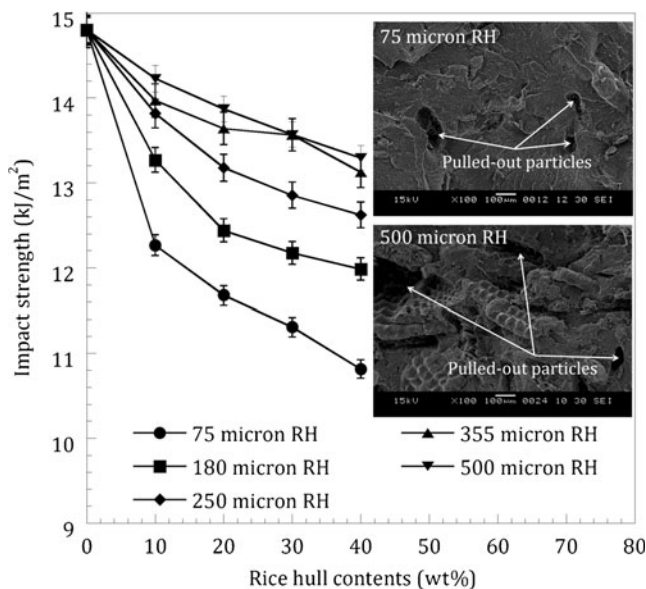

Fig. 1 Flexural strength of HDPE/RH composites with various RH particle sizes and contents
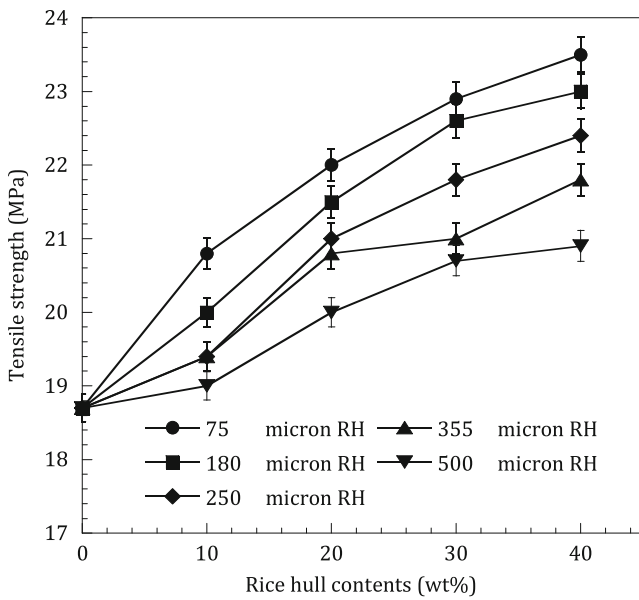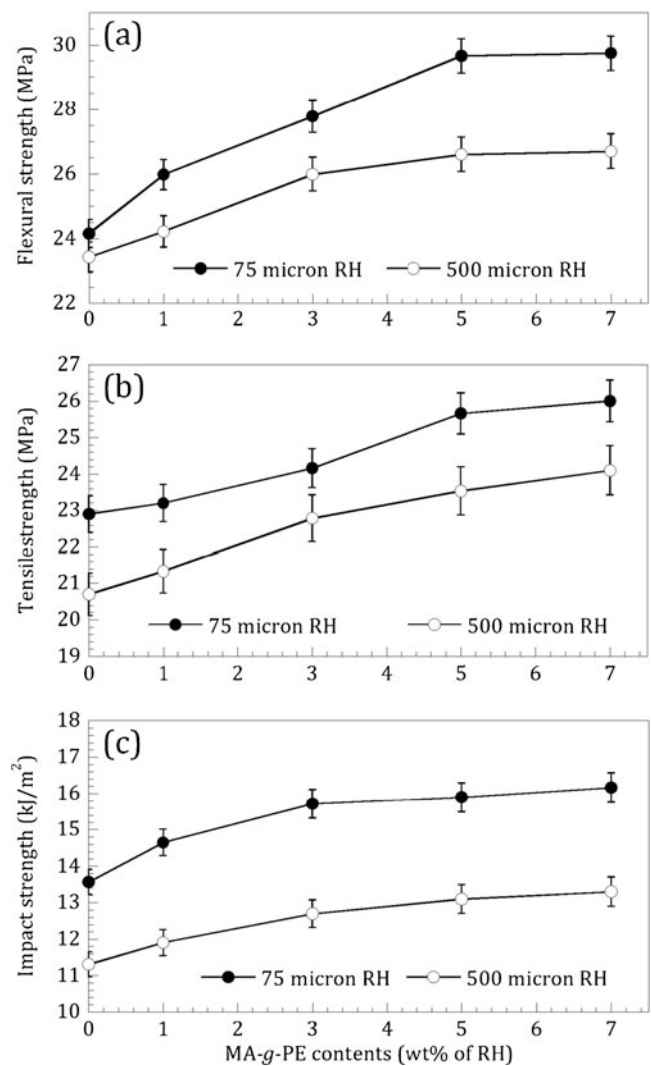
Fig. 2 Tensile strength of HDPE/RH composites with various RH particle sizes and contents

ASTM D 790 with a crosshead speed of 1.2 mm/min. Both the flexural and the tensile tests were carried out by using a universal testing machine (Instron, 5567).

The thermal degradation behaviors of all the HDPE/RH composites were characterized by using a thermogravimetric analyzer (TGA) (TA instruments, SDT Q600). Each specimen of the composites was heated at a controlled rate of 10 °C/min from 30 to 900 °C under nitrogen atmosphere. The TGA was conducted to study the decomposition behavior of the pure HDPE, pure RH, HDPE/RH composites and the HDPE/RH composites modified with MA-g-PE.

Fig. 3 Impact strength of HDPE/RH composites with various RH particle sizes and contents. Remnants of RH pulled out sites implied poor adhesion of the RH particles to the HDPE matrix

Fig. 4 Influences of the MA-g-PE compatibilizer on the mechanical properties of the HDPE/30 wt% RH composites with average RH particles of 75 and 500 μm **a** flexural strength, **b** tensile strength and **c** impact strength

Outdoor test was examined according to ASTM D 1435. The test specimen had no specific sizes. The pure HDPE and all the HDPE/RH composites were mounted on an outdoor rack during summer in Bangkok. The color changes of the HDPE/RH specimens after 90 days exposure were evaluated. The color measurement was conducted by using a color meter (BYK Gardness, Color flex) in CIE L* a* b* color system, in order to measure the change of color of the composites before and after natural weathering. Lightness (L*) and chromatic coordinates (a*) were measured for 5 replicate samples. L* is widely known as the reflecting diffuser, the maximum L* represents perfect reflecting diffuser or whiteness. The minimum value of L* is 0; it represents black. The a* and b* have no numerical limit, a positive a* indicates red and the negative one refer to green. The positive b* is yellow and the negative one is defined as blue. Thus, only L* and a* are enough for

indicating the coloration of the HDPE/RH composites in the current research.

The water absorption test was carried out according to ASTM D 570. The test method covers the determination of the relative rate of absorption of water by plastics when immersed in water at room temperature. This test was applied in the current study to investigate the absorption of water by HDPE/RH composites. The percentage increase in weight during immersion can be calculated by using Eq. 1 as follows:

$$\Delta M_t = \frac{m_t - m_0}{m_0} \times 100 \qquad (1)$$

where ΔM_t is the water uptake (%), m_0 and m_t are the mass of the specimen before and during aging respectively.

The morphology of the RH filler and the adhesion at interface between the RH phase and the HDPE matrix were investigated by using a scanning electron microscope (SEM) (JEOL, JSM 5800-LV) at an accelerating voltage of 15 kV. The impact fractured surface of each composite was mounted on an aluminum stub and sputter coated with a thin layer of gold before microscopic observations.

Results and discussions

Influences of the RH contents and their particle sizes on the mechanical performances of the composites

Figure 1 shows the effects of RH contents and its particle sizes on the flexural strength of the HDPE/RH composites.

The silica-rich content makes RH much more stiff and rigid than the soft HDPE matrix. The flexural strength was raised in all the composites regardless of the level of the RH compositions. The increment in the flexural strength may be due to the presence of silica-rich RH phase in the HDPE matrix. Furthermore, the results indicated that the particle sizes had no significant influences on the flexural strength. The result are in agreement with those reported by Cui et al. [24] that the flexural strength increased as the untreated wood fiber contents were raised for the weight fractions of wood fiber studied up to 50 wt%. A rather similar trend had also been reported for an increase of the flexural strength with greater wood particle content but still below 50 wt% [26].

The tensile strength of the HDPE/RH composites was graphically presented in Fig. 2. The incorporation of the rigid, silica-rich RH particles into the tough HDPE made the composite stiffer than pure HDPE. At 40 wt% RH, the tensile strength of HDPE/RH composites were all higher than that of the pure HDPE approximately 26 %, 23 %, 20 %, 17 % and 12 % for the average RH particles of 75, 180, 250, 355 and 500 μm RH respectively. It was observed that at the same RH fraction, smaller RH particles led to higher tensile strength. This was believed to be the consequence of small particles possessing larger surface area thus providing better transfer of load from the HDPE matrix to the RH phase.

Figure 3 illustrates the notched Izod impact strength of the HDPE/RH composites. The impact strength of pure HDPE was 14.80 kJ/m^2. All the HDPE/RH composites
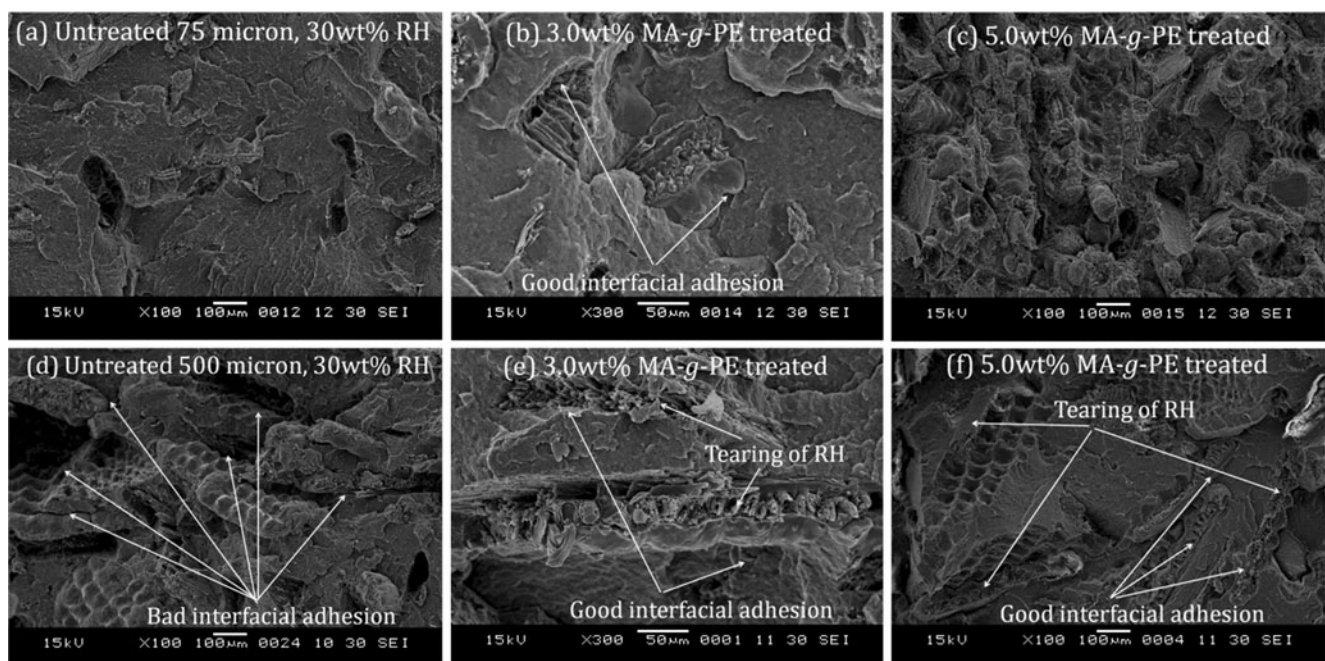

Fig. 5 SEM micrographs of the untreated and MA-g-PE treated HDPE/RH composites filled with average RH particles of 75 and 500 μm

exhibited lower impact strength than that of the pure HDPE. The HDPE/RH composites with the highest RH content possessed the lowest impact strength. For example, the impact energy of the HDPE/RH composites with 40 wt% of 75 μm milled RH particle was 10.83 kJ/m^2 while the HDPE/RH composites with the same RH content of 500 μm particle was 13.48 kJ/m^2. Nevertheless, they were still lower than that found in the pure HDPE approximately 27 % and 8.8 % respectively. The decline of the impact strength was believed to be the result from the RH reinforcing the matrix, causing the composites to become stiffer and more brittle. The ductile portion of the soft HDPE matrix was reduced, thus decreasing the composite toughness. Moreover, it was found that the composites with larger RH particles showed slightly higher impact strength than those with smaller particles. This is because smaller particles with its larger surface area tended to have more of the poorly-adhered interfacial sites, as indicated in the SEM micrographs shown in Fig. 3. The numerous partial separations or voids between the RH filler and the HDPE phase led to easier crack propagation along the interface upon impact [24, 25]. Numerous literatures had also reported that the impact strength of the untreated WPCs was reduced as the natural fiber contents increased in the HDPE/rice straw fiber composites [18], HDPE/bagasse composites [27], HDPE/pine wood composites [27] and HDPE/bamboo flour composites [28].

Influences of the MA-g-PE contents on the mechanical properties of the composites

As a remedy to the problem of poor interfacial adhesion between the HDPE matrix and the RH phase, the MA-g-PE compatibilizer was applied in the HDPE/30 wt% RH at the range from 0 to 7.0 wt%. Figure 4(a) shows the flexural strength of the HDPE/RH composites with 30 wt% of 75 and 500 μm RH treated with MA-g-PE ranging from 0 to 7.0 wt%. The flexural strengths of the composites with 75 and 500 μm RH without MA-g-PE were 24.16 and 23.43 MPa respectively. All the treated composites exhibited higher flexural strength compared with the untreated one. The HDPE/RH composites treated with 5.0 % MA-g-PE demonstrated an optimum enhancement in the flexural strength by 23 % and 14 % respectively. The addition of MA-g-PE in the HDPE/RH composites was expected to enhance better interfacial adhesion through the reaction between the maleic anhydride (MA) groups with the hydroxyl groups on the surface of RH to form strong covalent ester linkages, leading to a strong interfacial bonding between the RH filler and the HDPE matrix. The improved interfacial adhesion was confirmed by the SEM micrographs as demonstrated in Fig. 5. Figure 4 (b) compares the tensile strength of the WPC treated with MA-g-PE at identical RH contents and RH particle

sizes to those in Fig. 4(a). All the MA-g-PE treated WPC samples possessed higher tensile strength than the untreated ones. This implied that the compatibilized HDPE/RH composites could withstand higher tensile stress. Moreover, the MA-g-PE modified HDPE/RH composites also exhibited slight improvement in the impact strength, absorbing more energy upon impact by approximately 14 % and 18 % respectively when the compatibilizer was added at 7.0 wt%, as exhibited in Fig. 4(c). The higher mechanical strengths of the MA-g-PE modified HDPE/RH composites were results of better adhesion at the interface. The MA-g-PE treated HDPE/RH composites posed higher homogeneity and relatively higher than specimens without MA-g-PE. The improvements of all
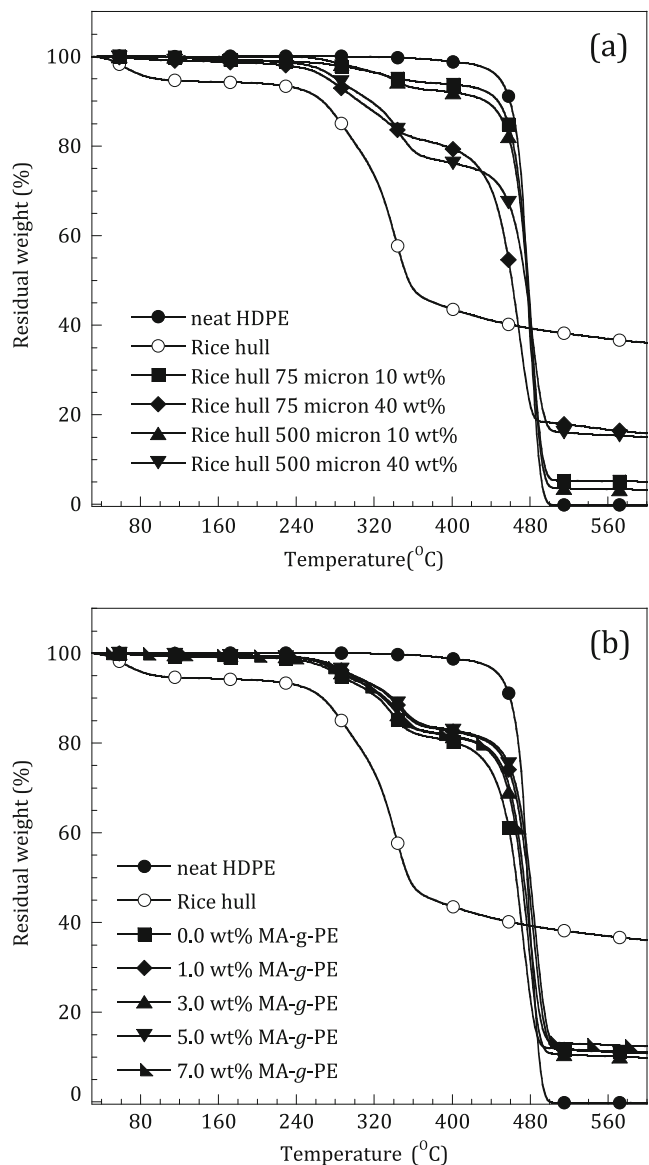
Fig. 6 TGA thermograms of the neat HDPE, RH and the HDPE/RH composites with various **a** RH contents and particle sizes and **b** MA-g-PE contents

flexural and tensile of the treated HDPE/natural fiber composites have been noted before [24, 28–30].

The morphology of the fractured surfaces of the HDPE/RH composites at different RH contents, various particle sizes with and without MA-g-PE was illustrated in Fig. 5. For the untreated HDPE/RH composites in Fig. 5(a) and (d), RH pulled-out was observed along with large amount of voids, implying the poor bonding interfacial area between the HDPE matrix and the RH filler. Compared to that with smaller RH particles, the composites with larger RH particle sizes showed numerous partial separations between phases. On the contrary, the HDPE/RH composites treated with MA-g-PE were seem microscopically to have improved HDPE/RH adhesion. It was evident from Fig. 5(b), (c), (e) and (f) that the treated RH was more uniformly coated by layers of the HDPE matrix, reducing the interfacial separation or voids. Remnants of torn RH particle were also found embedded in the HDPE matrix for all the MA-g-PE treated composites. They were morphological clues verifying that the MA-g-PE improved the interfacial adhesion. The improved interfacial adhesion, as was evident from the microscopic study, supported that the treated RH filler facilitated better stress transfers from the HDPE matrix, leading to the increment of the mechanical strengths of HDPE/RH composites.

Influences of the contents of the RH and the MA-g-PE on thermal degradation of the composites

Figure 6 shows the TGA thermograms of the pure HDPE, RH, untreated and treated HDPE/RH composites. The TGA scan indicated that the decomposition of RH occurred at the temperature range of 250–350 °C, corresponding to the decomposition of organic constituents within the RH such as cellulose, hemicellulose and lignin. The decomposition of RH left a char yield of 40 wt%. The decomposition of HDPE took place at the temperature ranged from 400 to 500 °C. Above 500 °C, the quantity of HDPE residue was very small due to the degradation of HDPE to gaseous products at higher temperature. Similarly, the HDPE/RH composites were found to decompose over two temperature ranges, i.e. 250–350 °C and 400–500 °C. The first thermal degradation corresponded to the decomposition of major constituents in the RH whereas the second degradation related to in the thermal decomposition of the pure HDPE. At the first decomposition range, the weight loss of the HDPE/RH composites decreased with greater RH content. Specifically, the weight loss of the HDPE/RH composites was 10 % and 25 % when the RH content was raised from 10 wt% to 40 wt% respectively. Furthermore, the char yield of the HDPE/RH composites increased with greater RH contents. The char yield of the HDPE/RH composites with 10 wt% and 40%wt RH contents were 18 % and 5 % respectively. The particle sizes of the milled RH had no significant influences on the degradation temperature of the composites, but it was found that larger RH particles lowered the char yield to around 2 % when compared with smaller particles. The TGA thermograms of the MA-g-PE treated HDPE/RH composites with 30 wt% of the 500 μm RH particles was shown in Fig. 6(b). The treated composites were found to also decompose

Fig. 7 Visual appearances of the HDPE/RH composites with 40 wt% RH and various RH average sizes, a neat HDPE b 75 μm, c 180 μm, d 250 μm, e 355 μm and f 500 μm before and after natural UV-weathering testing for 90 days
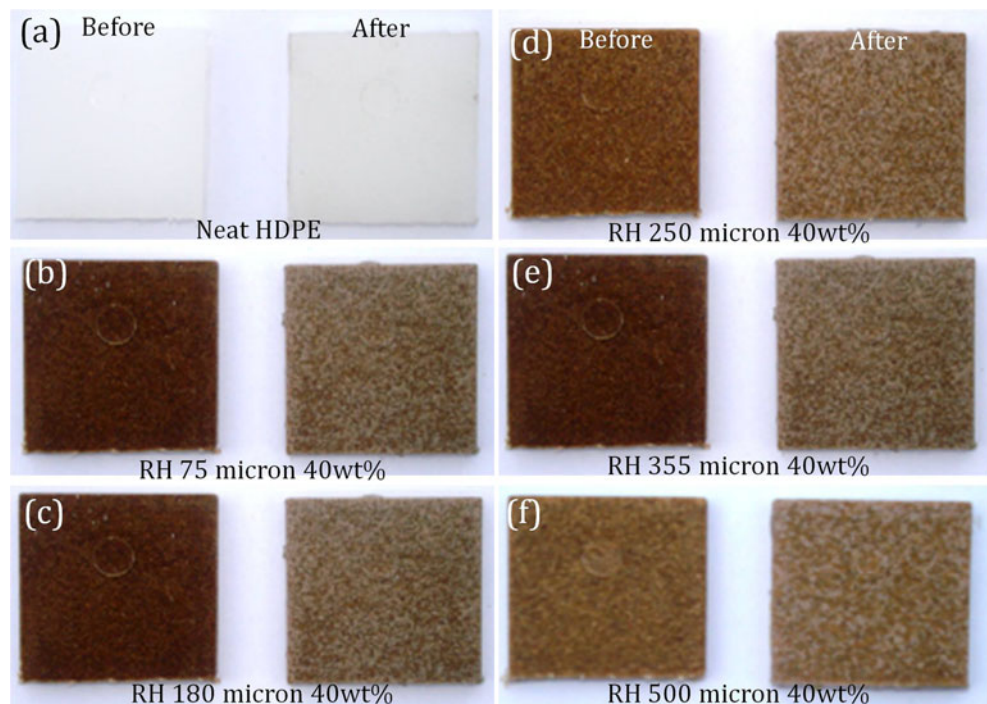
at two steps. The thermal stability and degradation temperature of HDPE/RH composites treated with MA-g-PE were slightly higher than those of untreated composites and were slightly increased with increasing MA-g-PE contents. The thermal stability of the treated composites was improved due to enhanced interfacial bonding.

Natural weathering test

In terms of appearance, the HDPE/RH composites with smaller RH particles appeared homogeneous with dark brown color while those with larger particles have a lighter shade of brown as shown in Fig. 7. The fine particle size of 75 μm functioned as a pigment in the HDPE/RH composites. The large RH particle such as at 500 μm, showed up as dark spots on the HDPE matrix. The influence of natural weathering on the visual appearances of the HDPE/RH composites was studied by exposing the HDPE/RH composites specimens to natural UV for 90 days. The exposed HDPE/RH composites were aged as illustrated in Fig. 7. The pure HDPE exhibited light yellow color after the 90 days of exposure. The weathered HDPE/RH composite clearly looked paler in color with greater surface roughness than the non-weathered ones. The corresponded colorations of all the composites in Fig. 7 were measured by L* and a* as clearly listed in Table 1. The appearance of the HDPE/RH composites over the range of the milled RH particle sizes studied appeared differently depending on the RH particle sizes. Before aging, the L* and a* values of the HDPE were 94.67 and 0.02 respectively, they implied that the unfilled HDPE color is white. Regardless of the RH particle sizes, the brown color of the RH was observed uniformly all over the entire surface compared to that of the unfilled HDPE with a decrement in the L* value. This indicated that the composites changed from light to darker shade when the RH was added. The increment in the a* confirmed that the composites were changed from white to the redder shade. Smaller RH particles tend to induce darker color than the larger ones. After natural aging for 90 days, both L* and a*
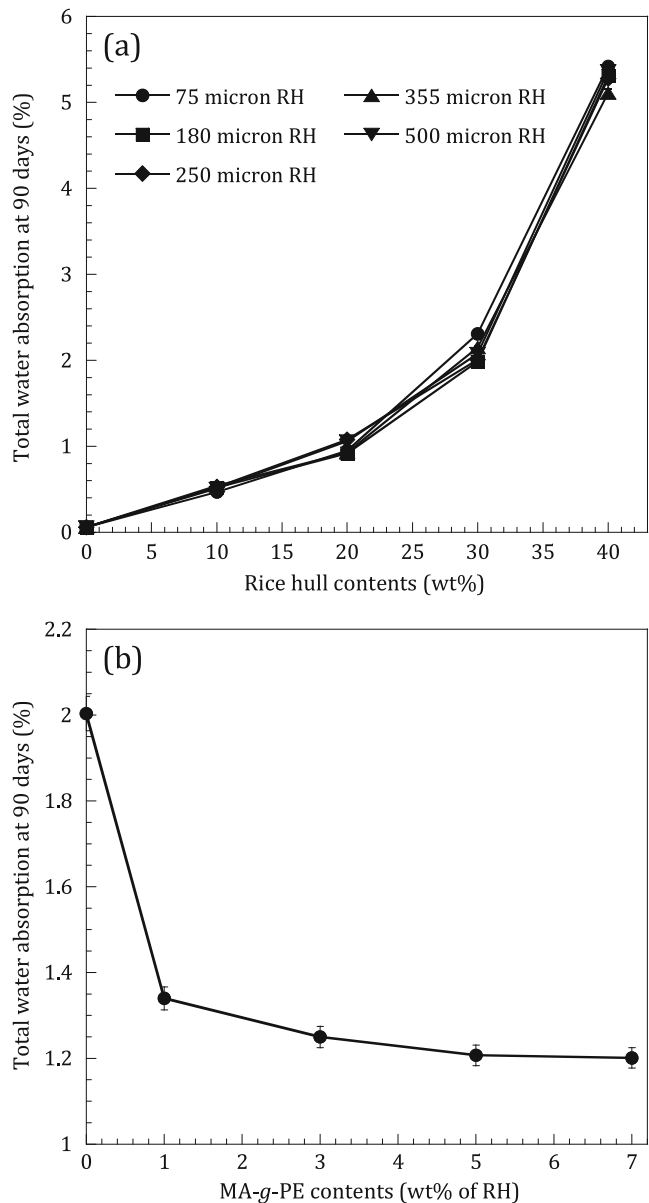
were significantly changed. The increments of L* values were indicated that the specimens were lighter nearly white corresponding to the decrement of the a* values. This implied that the composites were paler after the sun exposure as a consequence of UV degradation.

Water absorption of the HDPE/RH composites

The water absorption of the HDPE/RH composites at the soaking time of 90 days is demonstrated in Fig. 8(a). Hydrophobic HDPE absorbed water only 0.06 % while all the HDPE/RH composites showed high uptake of water. Hence, the water absorption of the HDPE/RH composites clearly

Table 1 Influences of the RH particle sizes and the natural weathering on the L* and a* value of the HDPE/40 wt% RH

Samples	Before natural weathering		After natural weathering	
	L*	a*	L*	a*
HDPE/75μm RH	51.45±0.12	4.72±0.23	72.19±0.14	2.01±0.11
HDPE/180μm RH	57.64±0.16	4.42±0.18	76.58±0.18	2.11±0.11
HDPE/250μm RH	59.90±0.21	4.05±0.15	76.35±0.15	2.06±0.10
HDPE/355μm RH	65.71±0.27	3.79±0.11	78.76±0.22	2.02±0.16
HDPE/500μm RH	69.52±0.19	3.07±0.22	77.33±0.14	2.12±0.24
Neat HDPE	94.67±0.14	0.02±0.00	93.54±0.16	0.03±0.00

Fig. 8 Total water absorption of HDPE/RH composites after immersion in the water for 90 days with various **a** RH particle sizes and contents and **b** MA-g-PE contents

increased with the increment of the RH contents. The hydroxyl group of the lignocellulosic constituent of the RH filler was responsible for the absorption of water. The size of the milled RH particles had no significant influence on the water absorption of the HDPE/RH composites. Figure 8(b) shows the water absorption of the HDPE/RH composites filled with 30 wt% of 500 μm RH particles treated with MA-g-PE from 1 to 7 wt%. The presence of the MA-g-PE compatibilizer evidently decreased water absorption of the modified HDPE/RH composites by approximately 33 %, 38 %, 40 % and 41 % when MA-g-PE was employed at 1, 3, 5 and 7 wt% respectively. As the MA-g-PE improved the interfacial bonding, they were less interfacial voids which generally arose due to the poor adhesion between the RH and the HDPE matrix. These voids, which tended to be numerous in the untreated HDPE/RH, were the sites accommodating the water or moisture absorbed. They were reduced significantly as the interfacial adhesion improved when MA-g-PE was employed, consequently leading to less water absorption by the MA-g-PE treated HDPE/RH composites.

Conclusions

The HDPE/RH composites were more brittle with the addition of the milled RH. Their flexural and the tensile strengths increased with the silica-rich RH contents. An improvement of all the flexural, tensile and impact strength was evident when MA-g-PE was added at around 5.0 wt% of RH. Microscopic observations revealed some RH pulled-out and numerous interfacial cavities within the untreated HDPE/RH composites. However, the interfacial adhesion was significantly improved when MA-g-PE was employed. Prolonged outdoor exposure to UV led to color fading. Water absorption was reduced when the MA-g-PE was incorporated in the HDPE/RH composites.

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